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1. Your reference

MGH/MG/P12700GB

2. Patent application number
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0303999.7

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

DEVRO PLC
MOODIESBURN
CHRYSTON
GLASGOW G69 0JE

Patents ADP number *(if you know it)*

If the applicant is a corporate body, give the country/state of its incorporation

UK (as per A/L 31/3/03)
DMS 3/4/03 7054794001

4. Title of the invention

COLLAGEN FILM

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

CRUIKSHANK & FAIRWEATHER
19 ROYAL EXCHANGE SQUARE
GLASGOW G1 3AE

Patents ADP number *(if you know it)*

547002

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Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

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Signature Date

CRUIKSHANK & RAIRWEATHER 21/02/03

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COLLAGEN FILM

The present invention relates to the use of porcine collagen derived from pigs, generally pig skin (also known as pig hide), for the production of a collagen film having improved properties. In particular, the collagen is derived from sows.

Artificial collagen films made from reconstituted collagen derived from natural animal sources have been commercially available for many years. These collagen films are typically used for wrapping food products, such as hams etc. At present, the principal source of animal collagen is bovine collagen derived from the hides of cattle. After the cattle have been slaughtered, the hides are removed and the underlayer composed principally of collagen is split away. The bovine collagen is then mechanically comminuted and formed into a gel in known manner. The gel is then extruded to form a film. The film is then cured, typically by change of pH and/or the use of cross-linking agents such as glutaraldehyde.

Collagen is also potentially available from a number of other sources, such as pigs, sheep, goats, avian, fish etc., but none of these have found widespread commercial use up to the present time. In particular, artificial collagen films and casings made from these sources, particularly porcine collagen, appear to have a number of disadvantages, particularly in having relatively low tensile strengths. Nonetheless, a porcine collagen film is currently available from Ed. Geistlich Sons Ltd., under the trade name Bio-Foil and is intended for wrapping hams.

It is, however, an object of the present invention to provide a porcine collagen film having improved properties.

In the prior art, attempts have been made to employ porcine-derived collagen, particularly collagen derived from pig intestines. Often, mixtures with bovine

collagen are employed. Thus, US 4,407,829, Sjolander discloses the use of pig intestines, pig lungs or cow rumen to produce a collagen slurry in a manner involving the use of proteolytic enzymes. US 5,411,887 Sjolander discloses the production of a collagen film through the enzyme treatment of pig intestines. US 5,840,849 Loders Croklaan discloses the use of a mixture of bovine collagen and pig intestine collagen treated with a proteolytic enzymes for the production of paste for co-extruded sausage casings.

Patent US 5,229,497 Teepak discloses the use of impure connective tissue derived from a variety of animal sources, including cattle poultry, swine and sheep, for the production of collagen casings. Skin and bone are excluded. The process involves the use of up to three enzyme treatment stages and the removal of fat from the connective tissue can be accomplished by a number of possible options. The only practical example disclosed involves the use of desinewed beef shanks.

There are also a number of prior art documents which involve the use of collagen derived from pig skins or hides. Patent US 4,196,223 Wilson Foods discloses the production of a collagen gel from pig skins and its subsequent coagulation and tanning to produce a collagen casing. However, the casing produced is said not to have adequate strength for use in commercial stuffing equipment (see US4,615,889). Patent US 4,615,889 Devro discloses the use of a mixture of bovine collagen and collagen derived from pork skin for the production of a collagen casing. Patent GB915441 Armour gives an example of the use of pig skins for the production of a collagen film.

The production of collagen films or casings by processes involving proteolytic enzymes are complex and consequently costly and may not have achieved commercial use for that reason. In some instances, mixtures of bovine and porcine

collagen have been used, presumably in order to achieve the necessary strength. In our experience, the production of collagen films or casings from purely porcine collagen leads to a product of poor tensile strength.

It is an object of the present invention to mitigate these problems and allow for the production of an essentially porcine collagen film.

The present invention is based on the discovery that the use of collagen derived from sow skins gives particular benefits.

Thus, one aspect of the present invention provides an extruded porcine collagen film made from an extrudable collagen gel; the collagen content of the film consisting essentially of sow collagen.

It has been found that the use of sow collagen imparts strength to the collagen film. In particular the collagen is present in the film substantially in the form of fibres. For this reason mechanical defatting of fibres (rather than chemical or enzymic processes) are preferred for preparing the extrudable gel. The fact that the collagen in the gel and in the finished film is derived from sows can be determined by Isothermal Shrink Tension (IST) measurements as described herein.

It is to be understood that typically the ratio of collagen to fat in natural pig hides or skins is in the region 1:1 to 1.5:1. The present invention preferably uses such sow skins or sow hides as the porcine collagen source. Preferably, the ratio of collagen to fat is at least 2.5 to 1.

The structural characteristics of pig skins are well known and are discussed for example in World Leather, October, 1997, page 85 – 90. Thus, pig skin is known to comprise from outside to inside an epidermis layer, dermis layer and subcutaneous fatty layer. The dermis layer is relatively thick compared to the epidermis and is the principal location of collagen fibres. The big bristles are also located in the dermis

layer and "cones" of fat tend to extend upwardly from the subcutaneous fatty layer through the dermis layer at the base of each bristle follicle. Thus, there tends to be a division between the collagen-containing dermis layer and the subcutaneous fatty layer. This division is less pronounced in younger pigs and more pronounced in older pigs.

There are a number of ways of reducing the ratio of collagen to fat in pig skins. One of the most effective ways is to carefully control the mechanical treatment of the pig skins in the tannery. The fresh pig skins can be subjected to mechanical defleshing which removes the subcutaneous fatty layer and some of the dermis layer to an extent that the ratio of collagen to fat is in the required ratio. Fat may also be chemically removed by treatment with alkali, such as sodium hydroxide. Smaller amounts of fat may also be removed at other stages during the preparation of the extrudable gel. Other options include removal of fat by solvent extraction (using acceptable food agents such as liquid carbon dioxide). Enzyme treatments are optional but not preferred since they appear to reduce the fibrous nature of the collagen.

In another aspect of the invention, the percentage of fat in the porcine collagen film is reduced to a level below 20%, particularly below 18% and especially below 16% by weight on a dry weight basis.

The ratio of collagen to fat is at least 2.5:1, preferably at least 3, particularly at least 3.5 and especially at least 4:1. Higher ratios of collagen to fat above 10:1, and even above 20:1 may be achieved. Preferably, the ratio is above 30:1 and especially above 40:1. However, the fat content is preferably controlled to achieve a good overall balance of properties in the final collagen film. Preferred ranges include 25:1 to 50:1, particularly 30:1 to 45:1. Thus, a certain proportion of fat in the final film

improves the appearance of the film, giving it an attractive sheen, and where the film is to be used around cooked products, tends to improve the cooking properties of the film. The unsaturated nature of the pig fat may provide unexpected strength (eg. via cross-linking). Thus, the amounts of other additives, such as glycerol or other humectants, included in the product may depend to an extent on the proportion of fat.

The object of the present invention is the provision of a collagen product essentially from porcine sources. The inclusion of bovine collagen is not preferred but minor amounts, preferably less than 10% and particularly less than 5%, of collagens derived from sheep, poultry, birds, fish etc., may optionally be included.

The collagen properties can be varied by mixing collagen derived from young pigs (about 4 months old) and sows (female pigs about 1 year old or more) in ratios of 0:100 to 10:90 (particularly 5:95). Older sow material tends to increase strength.

Apart from the required defatting, the porcine collagen may be processed in conventional manner to produce an extrudable aqueous gel. Generally, the porcine raw material is defatted and then disintegrated firstly in a mincing machine and secondly in a plate mill to produce a fibrous paste. Fat may be mechanically removed from the fibrous paste. The paste is then acidified with a strong mineral acid such as hydrochloric acid or with an organic acid such as lactic acid to swell the collagen. Alternatively, an alkaline swollen gel could be produced according to known techniques.

It is optional to include an alginate ester, such as an alginate glycol e.g. ethylene glycol alginate or propylene glycol alginate in the extrudable gel. This has been found to improve the strength, particularly the wet strength, of the film. Thus, improvements are found in the Burst Height Retention value and also in the Machine Direction (MD) wet tear strength. Generally, the alginate ester is present in the gel

in up to 1% by weight, preferably up to 0.5% by weight. In the gel and the film the ratio of collagen to alginate ester is generally in the range 95:5 to 75:25.

Other additives including humectants such as glycerol and sorbitol together with other desired known additives (e.g. flavours, colours and spices) may be included. The humectant is preferably present in the film in an amount of 15 to 45%, preferably 10 to 40% on a dry weight basis and may comprise glycerol, sorbitol or mixtures thereof. Cellulose may be included to modify the shrink tension of the film. The gel may also include coagulating agents such as minor amounts of glutaraldehyde, glyoxal, liquid smoke or multivalent cation (such as aluminium) which are effective to cross-link the collagen film and thereby increase its strength. Aluminium ions also waterproof the film. This increase in strength may, however, be at the expense of reduced elasticity. The gel is then homogenised, filtered and allowed to stand prior to extrusion.

Generally, the collagen solids content of the gel on a dry weight basis is in the range 2 to 10%; preferably 2.5 to 7%.

Extrusion is generally carried out through a slot extruder and the extruded material is generally applied onto a support belt to a wet thickness in the range 0.2 to 5 mm. The extruded film may be further treated with a liquid coagulating agent such as a salt bath (for example, sodium chloride or ammonium sulphate solution), an alkali bath (for example sodium carbonate) or a glutaraldehyde solution to coagulate the film. Coagulation may also be achieved using gaseous alkali such as ammonia gas. These treatments may be applied before or after drying the film.

Of course, the film could be further processed into other products. For example, it could be slit and twisted to form an edible string in known manner. The

string could be used to form netting. The string and netting may be used, for example, for trussing pork roast, shoulder, belly or hams.

The porcine collagen films of the present invention have been found to have a wet tear strength in the extrusion direction (MD) greater than 300 g/mm, particularly greater than 400 g/mm and often greater than 500 g/mm by the test methods disclosed herein.

The porcine collagen film has good strength, particularly in the dry state, and may be used for wrapping moist food product, such as pieces of pork or other meat. It is particularly useful for the production of shaped cooked hams from pork shoulder pieces. The porcine collagen film exhibits good strength, good cooking abilities, good appearance and film integrity. Thus, further aspects of the present invention include a process of producing the porcine collagen film, as well as a food product (especially a pork product) wrapped with the porcine collagen film or coated with the collagen gel and cured to form a film.

Embodiments of the present invention will now be described by way of example only.

Background

Commercially available (Ed. Geistlich Sons Ltd.) porcine collagen film (Geistlich 1and 2) have been analysed and found to contain the following on a dry weight basis:

	Geistlich 1	Geistlich 2
Collagen	48.7 %	68.8%
Glycerol	19.7%	20.6%
Fat	30.6%	12.4%

Ash	0.7%	0.4%
Collagen: Fat Ratio	1.6:1	5.5:1

Throughout these examples, the weight percentages will approximate 100% but within the limits of experimental error.

Defatting

The raw material is normally received as a salted pig (sow) skin (hide).

A typical defatting process would involve some or all of the following steps:

1. Initial Soak - Add the skins to the processing drum and add between 150% to 200% equivalent weight of fresh clean water at 28 deg C. Rotate for up to 1 hour and drain the vessel.
2. Main Soak - Add water (28 to 32 deg C) equivalent to 100% to 200% weight of raw hides. Add up to 0.5% of sodium carbonate or the like (helps to rehydrate through elevating the pH) and up to 0.2% by weight of wetting agent such as Danol WA (helps rehydration and removal of surface fats).
3. Fat removal - Remove hides from the vessel. Feed the whole hide pieces into a proprietary fleshing machine such as those made by Poletto, Rizzi, Mosconi & Persico. Set the cutter height to an appropriate position to effect good visual fat reduction without unduly removing good collagen. Fat removal can also be done prior to the soaking stages using a suitable proprietary fleshing machine. Hides prepared in such a manner are usually shipped in a salted condition but can also be frozen without use of salt. Material prepared in such a fashion can proceed to unhairing straight away after completing the initial soaking stages.
4. Unhair-Reweigh material into vessel. Add water (about 25 deg C) at up to 200% equivalent weight of hides. Add up to 3.5% by weight of sodium sulfide or up to 5% by weight of sodium hydrosulfide, and a wetting agent at up to 0.2% by

weight. A strong alkali is usually added, such as sodium hydroxide or lime to maintain the pH at 11 to 12 for the duration of the processing time. A liming auxiliary such as Erhavit MC or Aglutan PR at up to 0.3% by weight is usually added also. Typical processing times are between 12 hours and 60 hours before the liquor is drained.

5. Wash 1- Add fresh clean water (200% equivalent weight) along with a wetting agent (typically 0.2%) and rotate for 30 minutes then drain.

6. Wash 2 – Add fresh clean water (200% equivalent weight) and rotate for up to 30 minutes then drain. This stage can be repeated up to 4 times to remove residual surfactant (until no evidence of foam in the vessel).

7. Decalcification – Remove excess calcium ions (only where lime was previously used) with ammonium sulphate solution to a pH of around 9.

8. Buffer – Reduce pH of hides to around 2.5 to 6 with a solution of citric acid and sodium citrate, or hydrochloric acid.

9. Final washes – Wash hides with batches of fresh clean water to remove dissolved salts to a level where the drained liquor conductivity falls below 200μmhos.

Examples 1a & 1b

a) Salted sow skins were prepared by rehydrating, washing, unhairing & mechanically defatting using a method described above.

b) The resultant skin had a collagen:fat ratio of 44:1

c) The skins were then further washed, decalcified and buffered to a pH of around 4.5 and then washed again to reduce the level of dissolved salts.

d) These skins were next disintegrated; firstly with a mincing machine and then a plate mill to produce a fibrous paste.

e) This paste was blended together with a mixture of glycerol, sorbitol, hydrochloric acid, aluminium sulphate and glutaraldehyde.to form a swollen aqueous paste of constituents by weight (the balance being water);

HCl	0.12%
Collagen	3.03%
Fat	0.07%
Glycerol	1.16%
Sorbitol	0.29%
Aluminium ion	0.0106%
Glutaraldehyde	0.0012%

f) This paste was homogenised through a dairy homogeniser to produce a cohesive, smooth swollen gel.

g) This gel was extruded, through a slot extruder, to a wet thickness approximately 0.5mm, onto a continuous support belt and dried at a temperature around 60°C.

h) The resultant dried film was wound using a proprietary winding machine.

i) The film was further processed by treating with ammonia gas until the pH was increased to above 5 (Ex1a in the Table).

j) A further embodiment of the above film was manufactured from the same gel and extruder but treated with gaseous ammonia prior to drying and reeling (Ex 1b in the Table).

Examples 2a & 2b (PGA included)

a) Salted sow skins were prepared by rehydrating, washing, unhairing & mechanically defatting using a method described above.

b) The resultant skin had a collagen:fat ratio of 44:1

c) The skins were then further washed, decalcified and buffered to a pH of around 4.5 and then washed again to reduce the level of dissolved salts.

d) These skins were next disintegrated: firstly with a mincing machine and then a plate mill to produce a fibrous paste.

e) This paste was blended together with a mixture of glycerol, sorbitol, hydrochloric acid, aluminium sulphate, glutaraldehyde and propylene glycol alginate (PGA) to form a swollen aqueous paste of constituents by weight (the balance being water);

HCl	0.12%
Collagen	3.03%
Fat	0.07%
Glycerol	1.16%
Sorbitol	0.29%
PGA	0.3%
Aluminium ion	0.0106%
Glutaraldehyde	0.0012%

f) This paste was homogenised through a dairy homogeniser to produce a cohesive, smooth swollen gel.

g) This gel was extruded, through a slot extruder, to a wet thickness approximately 0.5mm, onto a continuous support belt and dried at a temperature around 60°C.

h) The resultant dried film was wound using a proprietary winding machine.

i) The film was further processed by treating with ammonia gas until the pH was increased to above 5 (Ex 2a in the Table).

j) A further embodiment of the above film was manufactured from the same gel and extruder but treated with gaseous ammonia prior to drying and reeling (Ex 2b in the Table).

TEST METHODS

The following test methods were used to obtain the data in the Table.

a) Weight

1. 1 metre of film is cut to length.
2. Film is weighed
3. Film width is measured
4. Weight per square meter is calculated

b) pH

1. A 5g sample of film is weighed into a mixing vessel.
2. 100 ml of distilled water at room temperature is added.
3. Film and water are blended together with a suitable high sheer mixer.
4. pH is measured with a suitable instrument after initially calibrating.

c) Burst height

1. Film is sampled at various positions across its width.
2. Each individual sample is clamped at the base of a perspex column of diameter 50.8mm
3. Water is introduced at a constant flow rate of 1.5L/minute to the top of the column
4. The height of water held before rupture is recorded
5. 15 samples (5 edge 1, 5 middle, 5 edge 2) are tested for each film type and the average is noted

d) Pierced burst height

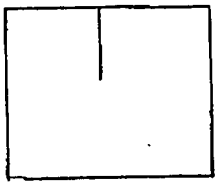
1. Test is conducted in same manner as burst height except that each film sample is pre-punctured by a 2-pin device with pins 15mm apart. The punctures are located near to the centre of the test area.

e) Burst height retention

1. A calculated value determined by dividing the average pierced burst height by the average burst height.

f) Wet tear test (both Md & Td directions)

1. Film samples are cut across the web to a size 9cm x 9cm.
2. A 4.5cm cut is made in the film in either the machine or transverse direction from the middle of an edge to the centre.



3. Machine and transverse directions are tested separately
4. The uncut portion of the samples are immersed in iced water for 30 seconds
5. The cut pieces are clamped in an Instron 5544 tensometer using the upper and lower jaw sets
6. Samples are pulled apart at a speed of 5cm/minute for a total distance of 4cm
7. Energy to tear is recorded

g) Colour

1. A proprietary colourmeter is calibrated using a suitable white reference tile.

2. 5 Test specimens of film are placed in turn on the reference tile and the colour is measured
3. The average L, a & b value is recorded.

h) <u>2D tear test both Md and Td directions</u>

1. Samples of film are cut to dimensions 19cmx19cm
2. They are next conditioned at 65% relative humidity and 20^0C for 24 hours.
3. Samples are stretched biaxially at a speed of 1cm/minute until break point.
4. The stress at break values are recorded
5. From the same tests the secant modulus at 2% stretch is calculated. (This indicates the elasticity of the sample at the very start of the test)

i) <u>Film Odour</u>

1. A test panel of people were asked to describe the odour and score it as Good, Fair or Poor.

j) <u>Appearance</u>

1. Visual colour/appearance of reeled film.

k) <u>Thickness</u>

Is measured with a proprietary thin film thickness measurement device e.g. an Elco meter.

Isothermal Shrink Tension (IST) determination

It is well understood that Mammalian collagen, in contact with water, contracts sharply at temperatures from 60°C - 70°C to about 25% to 33% of its initial length. If the shrinkage is inhibited, by rigidly mounting the sample, then considerable tension will develop. IST testing studies the thermal shrinkage of collagen by measuring the tension generated with constant rate heating,

The apparatus essentially consists of:

a) A pair of opposing jaws across which a test sample can be mounted, one set of jaws is directly connected to a strain gauge.

b) The sets of jaws are themselves mounted on a rigid reinforced frame.

c) Within a single frame a plurality of jaw sets can be attached. Most usually circumferentially and equidistantly.

d) A tank for immersing the whole frame and filled with a heating fluid, usually water.

e) A heater facility for the immersion tank to raise the temperature of the fluid.

f) A device for recording the tension generated over the time period of the test

g) A device for charting the output from the individual strain gauges.

A) Raw Material IST

The method consists of:

1) Stamping 5 test pieces of equal dimensions from the middle of the hide close to the spine, in the direction of the spine and with no visible fat present.

2) Checking the weight of every sample and then mounting them in each jaw set on the apparatus and taking care to securely fasten whilst also minimising residual tension.

3) Immersing the apparatus in the water bath and ensuring that all the test samples are fully immersed for the duration of the test.

4) Pre-heating the water in the bath to 40 °C

5) Setting all strain gauges to zero.

6) Commencing recording of tension generated from each jaw set.

7) Raising the temperature of the water bath at 1 °C per minute up to 90 °C

8) On completion of the test an equivalent untested sample set is analysed for collagen content.

9) The final tension data is averaged for the 5 samples and normalised back to the dry weight of material and then charted against temperature.

10) It is more usual to normalise the tension data back to dry collagen content derived from the material analysis.

11) For the examples disclosed the samples were stamped from buffered and washed hides, immediately prior to mincing and disintegration. IST testing can also be conducted on materials at other suitable stages of the process.

B) Film IST

The method consists of:

1) Stamping test pieces of equal dimensions across the full width of the film sample and in both the extrusion direction (Md) and the transverse direction (Td).

2) Each test comprises loading 32 test pieces into each jaw set and following the procedure set out above for the raw material tests.

3) After conducting the test, the tension data is averaged over the total number of individual tests.

4) Where the collagen content of the film samples are significantly different the data is normalised back to dry collagen content before charting against temperature.

Table

Test	Ex1a	Ex 1b	Ex 2a	Ex 2b	Geistlich 1	Geistlich 2
					Commercial Porcine Film	Commercial Porcine Film
Weight[a]	31g/m^2	31g/m^2	40g/m^2	40g/m^2	38g/m^2	35.5g/m^2
pH[b]	7.5	5.0	5.0	5.2	3.9	7.18
Burst Height[c]	47.1cm	44.1cm	58.7cm	57.3cm	66.6cm	69.4cm
Pierced Burst Height[d]	41.0cm	44.1cm	47.6cm	57.3cm	40.1cm	53.0cm
Burst height retention[e]	87.0%	100%	81.1%	100%	60.2%	76.4%
Wet MD tear test (total energy)[f]	416gf/mm	386gf/mm	505gf/mm	599gf/mm	205gf/mm	280gf/mm
Wet TD tear test (total energy)	324gf/mm	401gf/mm	322gf/mm	391gf/mm	210gf/mm	323gf/mm
Colour (L,a,b)[g]	97.05 -0.34 3.50	97.17 -0.17 2.92	96.78 -0.17 3.14	97.05 -0.30 3.68	96.49 -1.24 7.72	97.04 -0.81 6.13
2 D tear test Md stress at break (MPa)[k]	9.95±0.65	9.80±1.27	11.37±0.22	13.98±0.66	9.56±3.95	XXXX
2 D tear test Td stress at break (MPa)[l]	9.66±0.80	9.08±1.38	10.44±0.76	13.34±0.86	7.77±2.87	XXXX
2 D tear test Md 2% secant modulus (Mpa)[n]	104.87±17	109.33±22	183.83±7	100.67±24	119±20	XXXX
2 D tear test Td 2% secant modulus (MPa)[n]	139.06±21	131.83±27	198.50±7	140.50±39	164.25±36	XXXX
Film odour[i]	Fair	Good	Fair	Good	Poor	Poor
Appearance[i]	Pale straw	Pale straw	Pale straw	Pale straw	Straw	Straw
Thickness[k] (0.001")	0.87	0.86	1.24	1.24	1.04	1.01

Geistlich 1 and 2 are two samples of prior art film sold by Ed. Geistlich Sons Ltd. for comparison.

In the attached drawings:

Figures 1 and 2 show IST values in the machine extrusion direction (MD) and the transverse direction (TD) for the Devro films of the Examples (invention) and Geistlich 1 and 2 commercial porcine films for comparison;

Figures 3 and 4 show details thereof which illustrate more clearly the onset of shrinkage temperatures;

Figure 5 show IST values for the raw sow collagen material in comparison with raw pig collagen;

Figures 6 to 10 are scanning electromicrographs (SEM's) of the products of Examples 1a,1b, 2a, 2b and Geistlich (comparison) showing the fibrous nature of the films of the invention.

IST Chart Notes

Attribute	Devro films	Commercial porcine films
Md Shrinkage onset temperature	>52^{0}C	< 50^{0}C
Md Peak Shrinkage force	>90g	<80g
Md temperature at peak shrinkage force	>75^{0}C	<68^{0}C
Td Shrinkage onset temperature	>52^{0}C	<50^{0}C
Td Peak Shrinkage force	>50g	<65g
Td temperature at peak shrinkage force	>72^{0}C	<66^{0}C

In all cases the charts highlight that the Devro films have higher shrinkage onset temperatures, typically higher than 52^{0}C. For Geistlich films this value is below 50^{0}C.

The Devro films, in general, have considerably higher shrinkage tensions and the temperature of maximum shrinkage is also considerably higher.

This indicates the presence of a more thermally stable collagen.

Differential Scanning Calorimetry

DSC was carried out on the films by conventional techniques. The results for the films are given in the Table 2 below.

Table 2

Ref no	Ts (peak) - ^{0}C	Ts (onset)- ^{0}C	Energy (J/g of dry material)	% collagen	Energy (J/g of collagen)	Average
Geistlich 1	49.0	34.9	19.31	62.8649	30.7167	
Geistlich 1	48.3	35.1	19.61	63.2973	30.9808	30.8487
Ex 1b	66.2	47.8	44.73	61.7	72.5142	
Ex 1b	66.4	47.7	43.34	61.7	70.2608	71.3875
Ex 2b	65.3	54.2	28.00	58.3	48.0337	
Ex 2b	67.8	53.9	30.37	58.3	52.0995	50.0666
Ex 2a	61.7	49.6	25.72	59.5	43.2539	
Ex 2a	50.7	40.0	25.57	59.5	43.0017	43.1278

Examples 3 (Final Product Manufacture)

i) A mixture of the following is prepared and tumbled:

Pork Shoulder pieces	55Kg
Nitrite Salt	1.65Kg
Polyphosphate	0.25Kg
Ascorbate	0.08Kg
Dextrose	0.58Kg
Water	24.9Kg

ii) 1kg nominal weight hams are produced by applying elastic netting together with the collagen film of Examples 1a,1b,2a and 2b above using a proprietary ham production device such as those made by Possenti. The ends are clipped and the uncooked product is placed on a cooking rack. Air bubbles are allowed to escape by puncturing the film at appropriate places.

iii) The hams are cooked via a conventional process such as:

Cook	no smoke	20minutes	122deg F Dry bulb	100%RH
Cook	no smoke	30minutes	131degF Dry bulb	15%RH
Cook	smoke	30minutes	140degF Dry bulb	64%RH
Cook	no smoke	20minutes	140degF Dry bulb	15%RH
Cook	smoke	30minutes	149degF Dry bulb	64%RH
Cook	no smoke	20minutes	149degF Dry bulb	15%RH
Cook	smoke	30minutes	154degF Dry bulb	65%RH
Cook	no smoke	20minutes	172degF Dry bulb	100%RH
Cold Water Shower		20minutes		

The final product properties are set out in Table 3.

Table 3

	Stuffing/Clipping	Cooking	Appearance	Film Integrity
Ex 1a	Good	Good	Good	Good
Ex 1b	Good	Good	Good	Good
Ex 2a	Good	Good	Good	Good
Ex 2b	Good	Good	Good	Good
Geistlich 1	Good	Good	Good	Good
Geistlich 2	Good	Good	Good	Good

CLAIMS

1. An extruded porcine collagen film made from an extrudable collagen gel; the collagen content of the film consisting essentially of sow collagen.

Figure 1

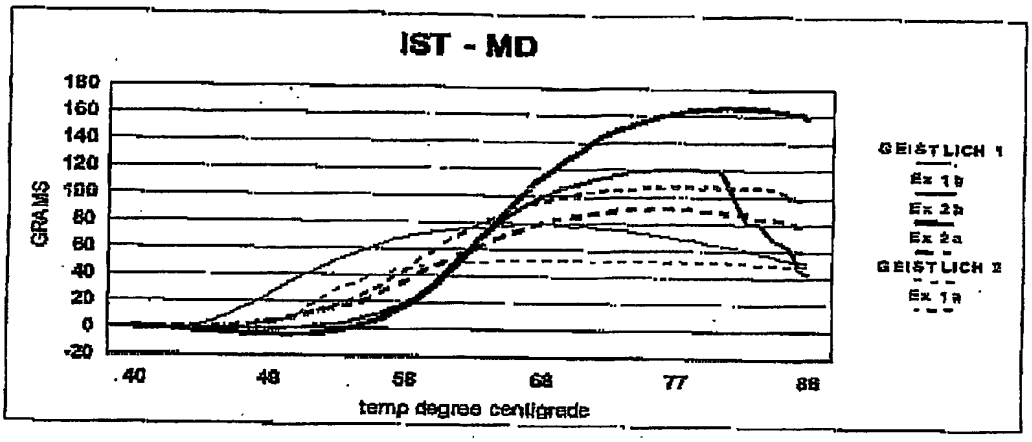


Figure 2

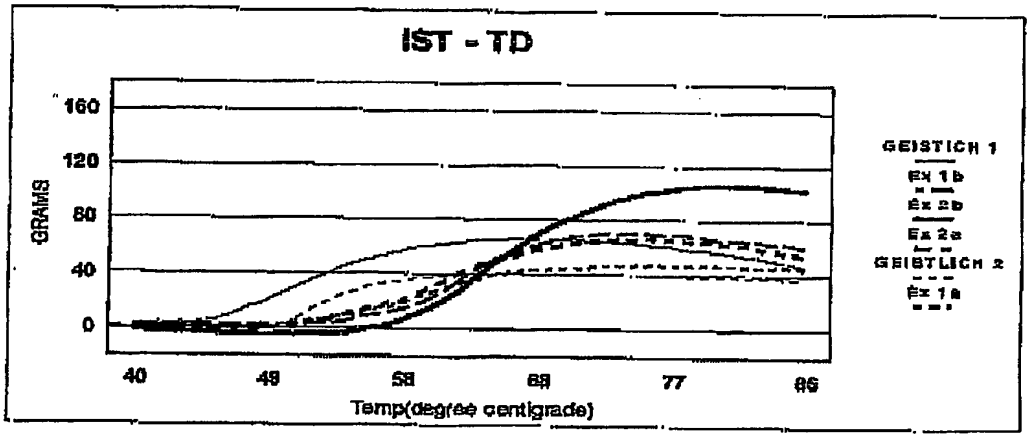


Figure 3

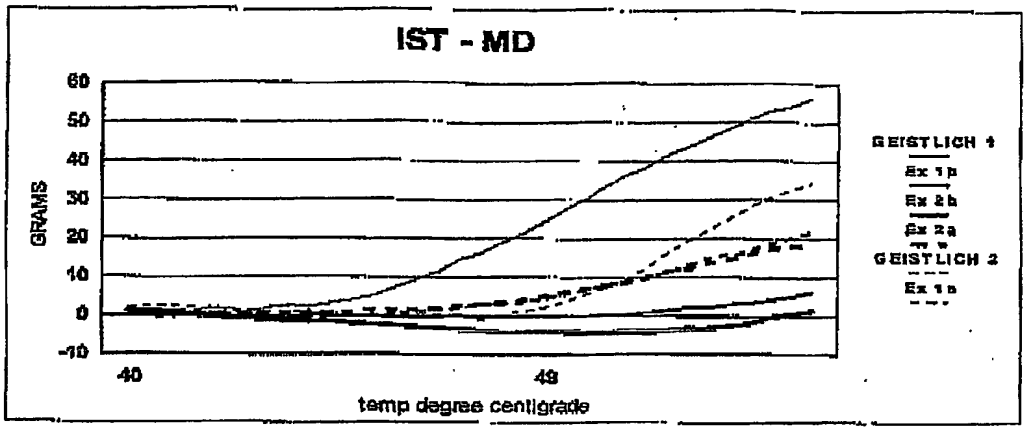


Figure 4

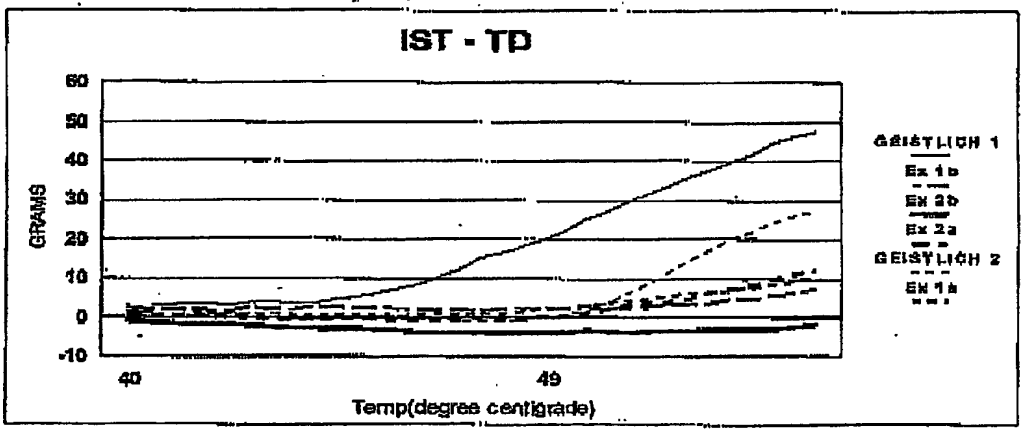
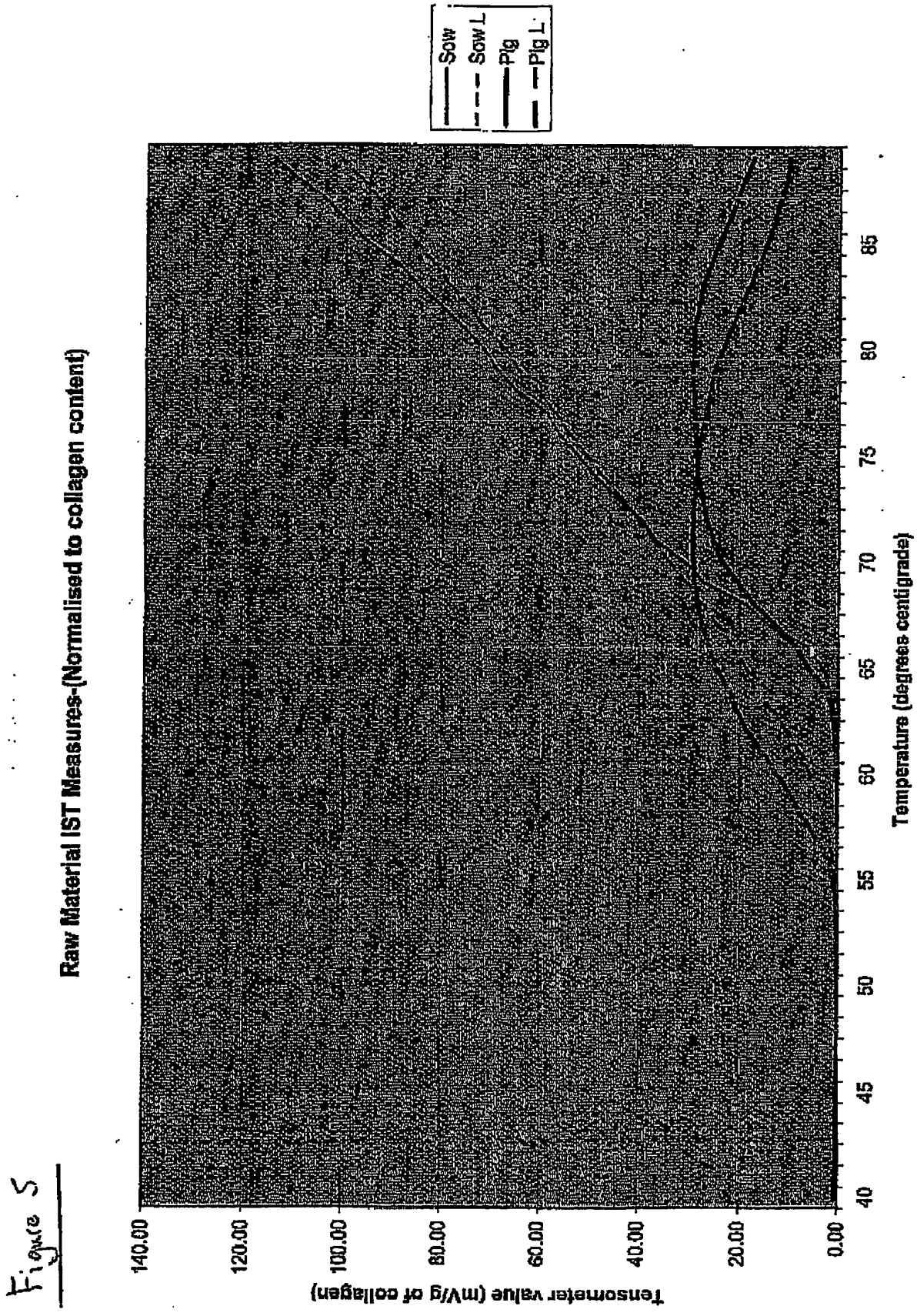


Figure 5


Raw Material IST Measures-(Normalised to collagen content)
Sow
Sow L
Pig
Pig L
Tensometer value (mV/g of collagen)
140.00
120.00
100.00
80.00
60.00
40.00
20.00
0.00
40
45
50
55
60
65
70
75
80
85
Temperature (degrees centigrade)

Ex 1a @ 1500 magnification

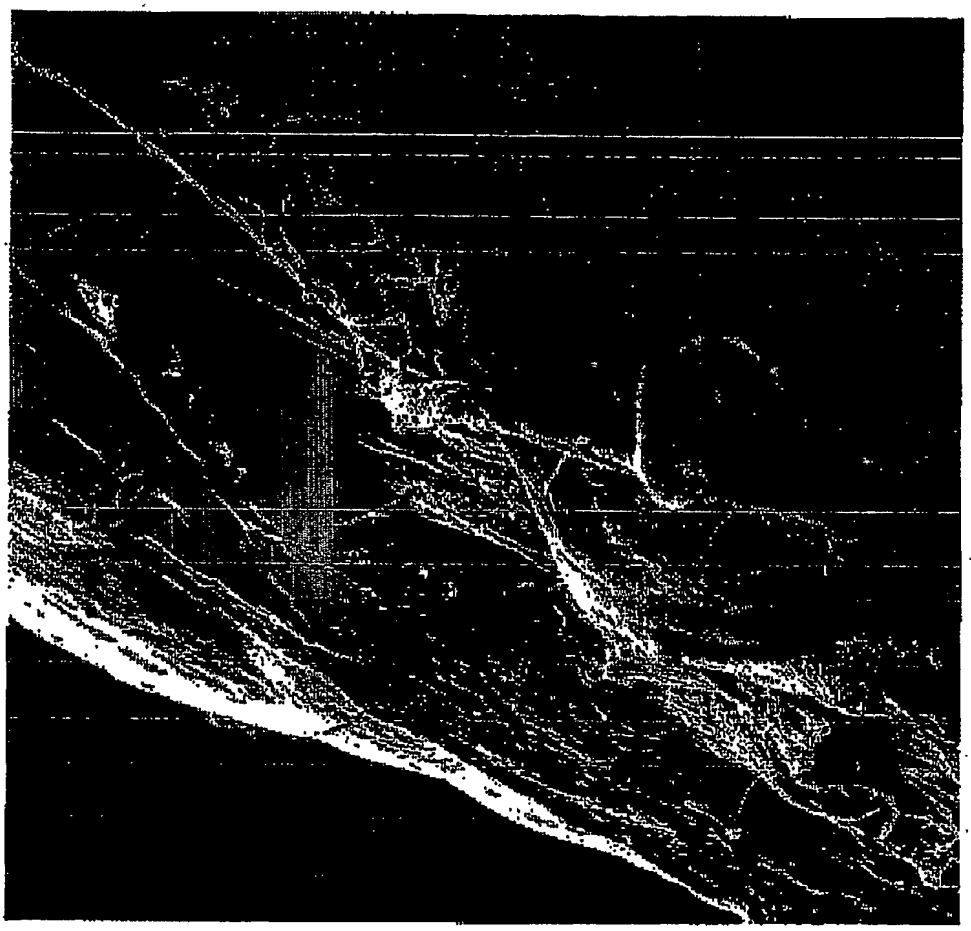


Figure 6

Fig 6

Figure 1

Ex 1b @ 1500 magnification

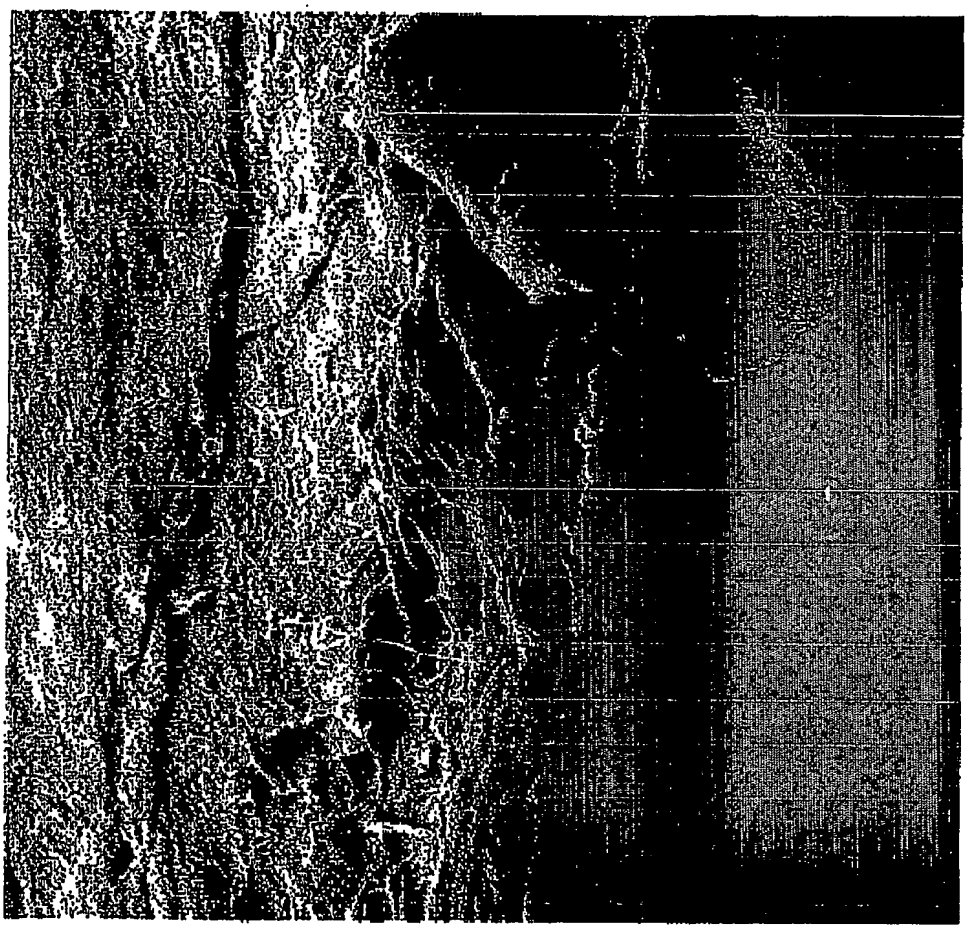


Fig1

Figure 8

Ex 2a @ 1500 magnification

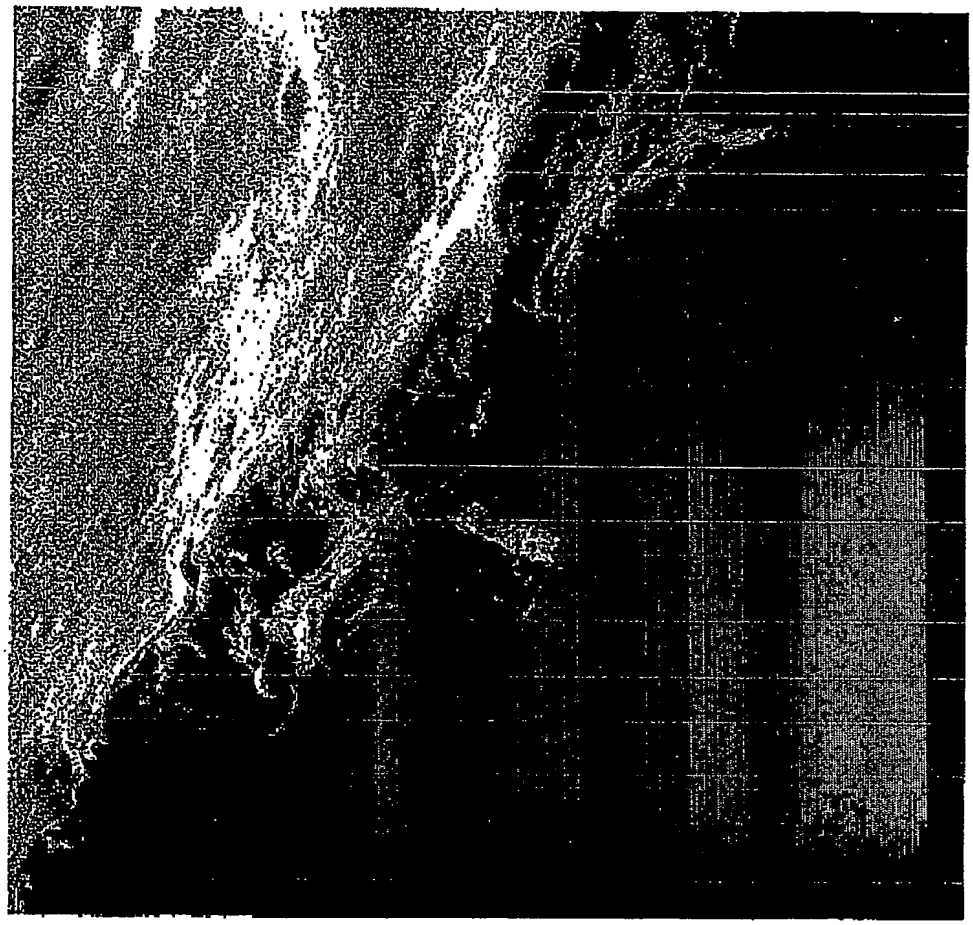


Fig 8

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Figure 9

Ex 2b @ 1500 magnification

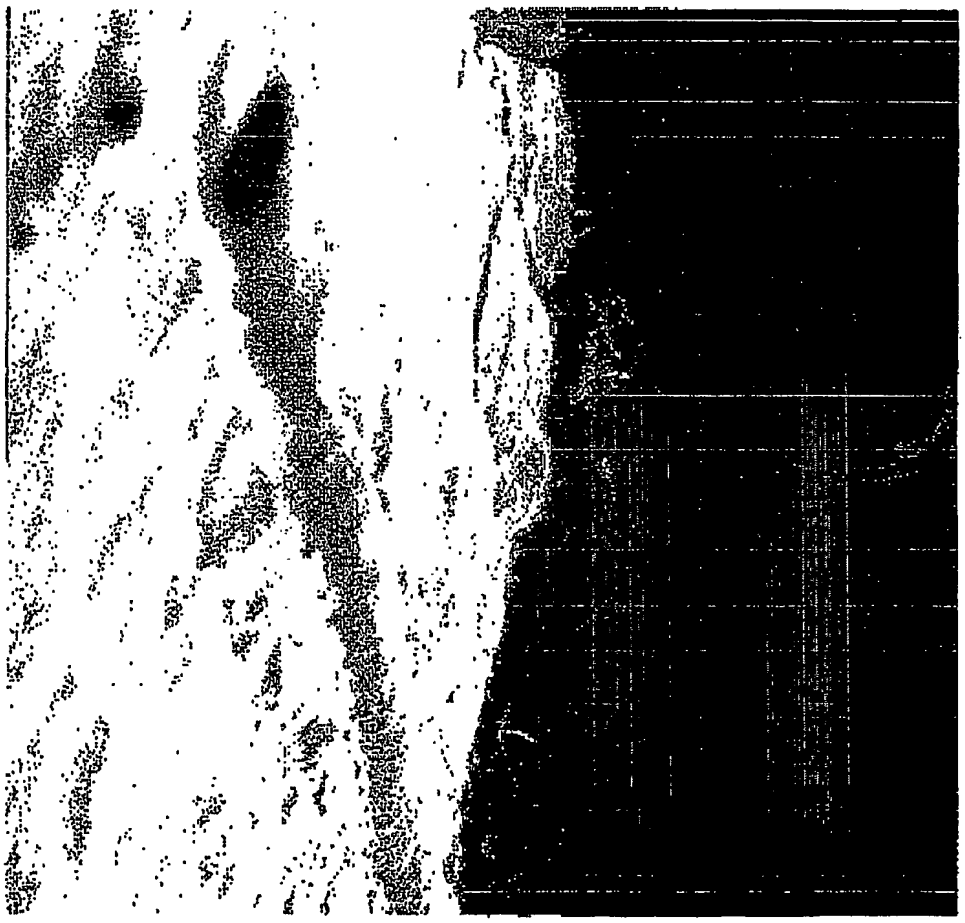

Fig 6

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Figure 10

Geistlich 1 @ 1500 magnification

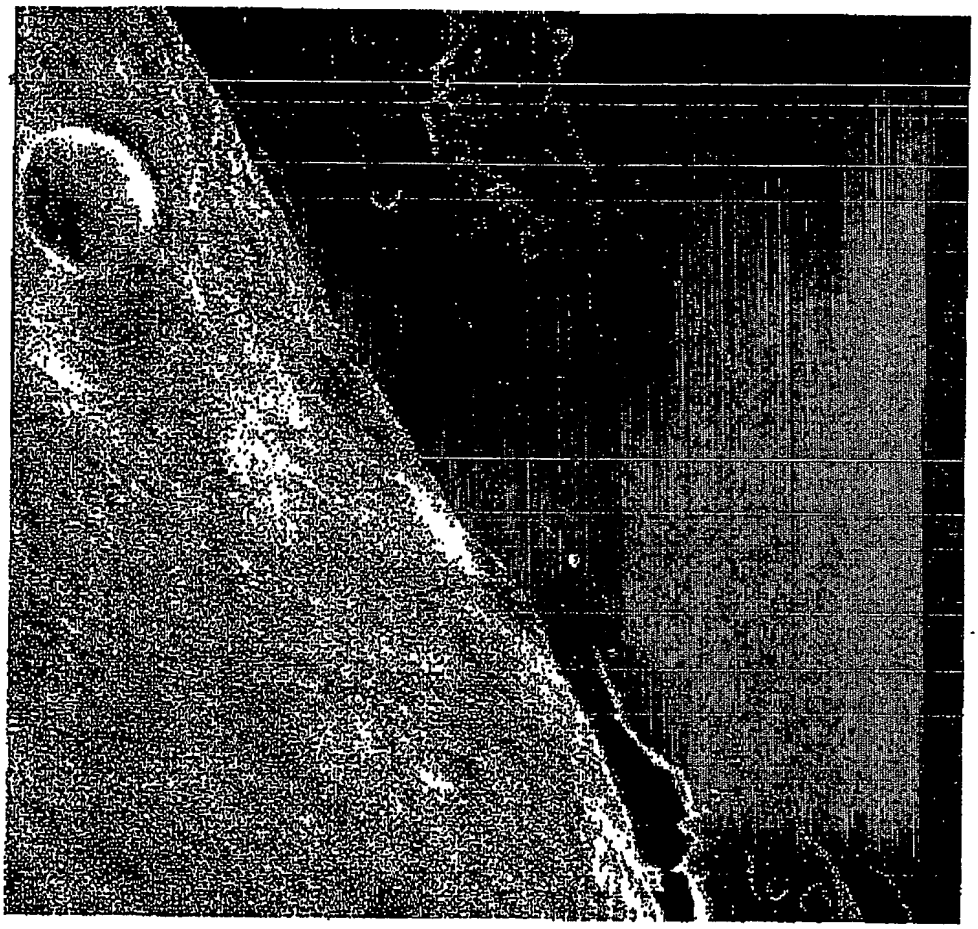

Fig 10

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Figure 10

Geistlich 2 @ 1500 magnification

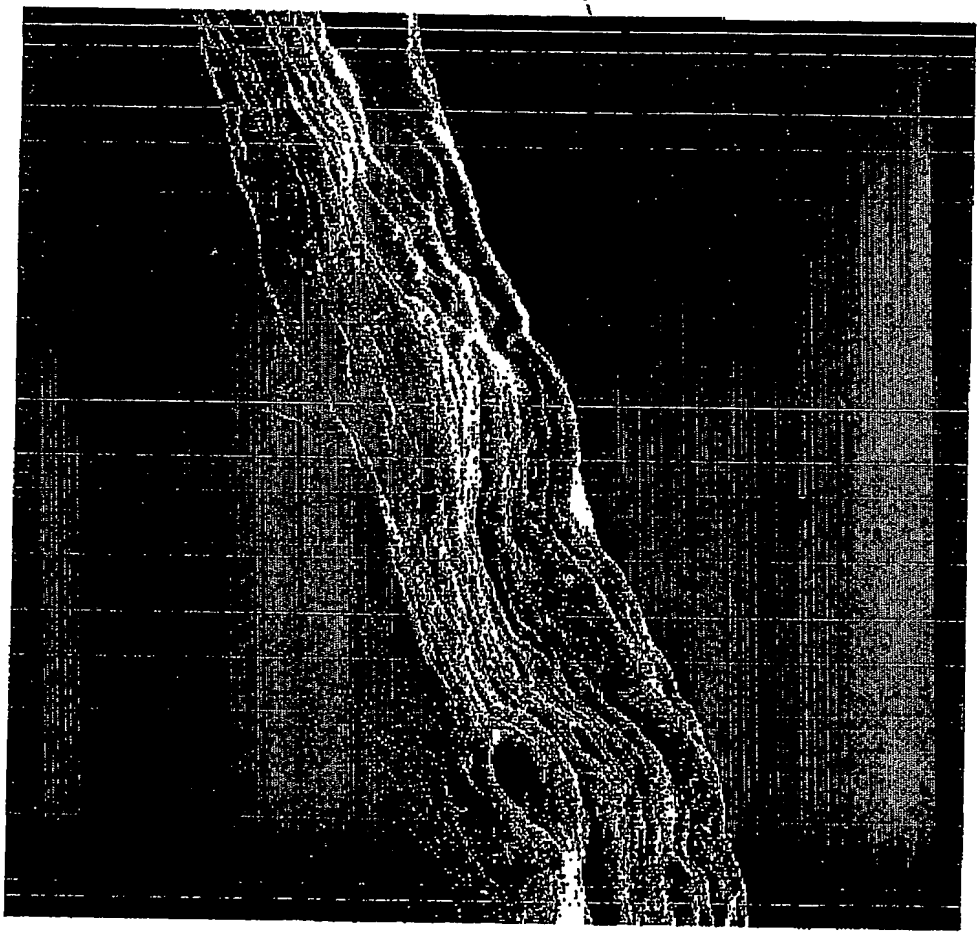
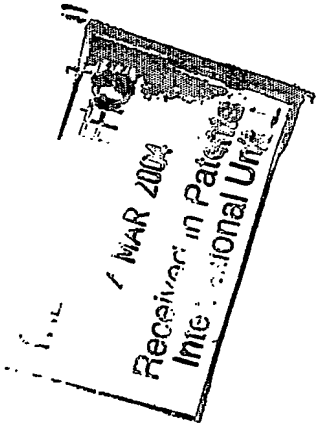
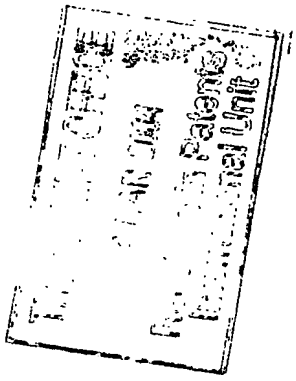


Fig 16





PCT Application
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